Characterisation of Fly Ash & Slag and Application of Alkali Activated Fly Ash in Pavement

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Characterisation of Fly Ash & Slag and Application of Alkali Activated Fly Ash in Pavement

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This is to certify that the thesis mentioned above submitted by Aishvary Mishra (215CE3277) in the partial fulfilment of the requirement for the degree of *Master of Technology* in *Civil Engineering* with specialization in *Transportation Engineering* at National Institute of Technology Rourkela is a reliable work carried out by him under my supervision and guidance.

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I, *Aishvary Mishra*, Roll Number *215CE3277* hereby declare that this thesis entitled Characterisation of Fly Ash & Slag and Application of Alkali Activated Fly Ash in Pavement presents my original work carried out as a post graduate student of NIT Rourkela and, to the best of my knowledge, contains no material previously published or written by another person, nor any material presented by me for the award of any degree or diploma of NIT Rourkela or any other institution. Any contribution made to this research by others, with whom I have worked at NIT Rourkela or elsewhere, is explicitly acknowledged in the dissertation. Works of other authors cited in this dissertation have been duly acknowledged under the sections “Reference”.

I am fully aware that in case of any non-compliance detected in future, the Senate of NIT Rourkela may withdraw the degree awarded to me on the basis of the present dissertation.

May 22, 2017 *Aishvary Mishra*

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Abstract

Every year, large amount of coal is using in different type of industries which is producing huge amount of by-products. Dumping of these by-products became a major issue. It required large piece of land, proper construction of dumping pit, high maintenance etc. similarly, in road construction, huge amount of aggregate is required. Instead of using natural aggregate, using of by-products can solve two major problems. One is, save land from dumping or digging, which can use in some other purpose and other one, save environment. But, before using of these by-products, proper analysis is required to expand the ways to reuse it firmly. For that purpose, information about not only of chemical composition of elements, but also chemical structure of fa and slag is required. Various properties like chemical composition, phase composition, shape and size, unburned carbon content, free lime content, pH value and toxic or hazardous elements present in fa and slag can be determined by using experimental methods like x-ray diffraction(XRD), titration method, Scanning Electron Microscope test (SEM), Electron Dispersive X-ray Spectroscopy (EDX) and Loss On Ignition test. Geopolymerization was done using sodium hydroxide with different percentage of NaOH.

Keywords: fly ash, composition, phases, unburnt carbon and lime content, geopolymer, unconfined compressive strength.

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Chapter 1

Introduction

1.1 Background of the study

Indian road network is the second largest road network in the world with total length of 55.32 lakhs Kms approximately. In that, length of national highways is around 1 lakh kms. Road network is very important for any country for their economic, social and industrial development. Indian roads transport total 85% of passengers and 70% of freight traffic of whole country. Indian government started National Highways Development Project to increase highway network and upgrade the state highways to provide fast transportation for agricultural and industrial sector of the country.[National highways]

Pavement is basically three types- flexible pavement, rigid pavement and semi-rigid pavement. In India, most of the roads are constructed as flexible pavement. But, from 2017, Indian government has decided the construction all new roads by cement concrete. Flexible pavements are constructed of bituminous and granular materials. Intensity of stresses in the flexible pavement is high at top so, better materials are used on top rather than bottom portion where stresses are less. Rigid pavements are constructed by Portland cement concrete. Mostly of the rigid pavement consist only three layers- PCC course, base/subbase course and subgrade. Sometime, if subgrade has sufficient strength, than base/subbase course can remove and PCC layer can directly laid on subgrade course. Semi-rigid pavements have properties of both type of pavement. Strength of this type of pavement is like concrete roads and maintenance is like flexible roads.

Like road network, industries are also a main component to develop a country. India is a developing country and Industries are the main source of economy for a developing country. Most important industrial sectors are metal extraction plants, power plants, petroleum refineries and so on. But for continuous working of these plants, coal is required.

1.2 Problem statement

Every year, a large amount of natural aggregates are used in construction of pavements. For the construction of base and subbase course, top layer of soil is also used which affect the agriculture strength of soil. Because of continuous digging of earth for getting aggregate, natural resources are reached at their end. We have to find some other alternatives to minimize the use of natural aggregate.

At the same time, because of burning of coal, large amount of fa generate. In year 2015-16, total 177 million tons coal generate in India but in that only 107 million tons utilized in different field, which is about 61 % of total generation. Fa is a hazardous material. It required a large piece of land to store and proper protection is required to prevent mixing of it to ground water. So, we have to find more area for the maximum utilization of fa.

1.3 Objective

The main objectives of this study are

- Structural characterization and chemical composition of different fa samples.
- Determination of presence and amount of hazardous materials such as heavy materials/ trace elements/ contaminants present in different fa samples being used to assess the harmful effects if any.
- Understand the effect of alkali activator like sodium hydroxide (NaOH) on fa of different sources by determining the compressing strength and CBR value.

After doing the structural characterization and chemical composition, it is easy to understand the behaviour of fa in different engineering applications and the presence and extent of hazardous constituents such as heavy metals/ contaminants/ trace elements.

Chapter 2

Literature review

2.1 Chemical composition of Fly ash

In fa, about 90 % of the particles are non-crystalline. This is because of rapid cooling of fa when it extract from the flue gasses. Major crystalline particles in fa are quartz, mullite, hematite and magnetite. Silica in fa present in both form- crystalline and non-crystalline. Reactivity of fa is basically depend on how much non crystalline particles present. In India, most of the coal is bituminous and lignite. So, maximum amount of fly is class F type. Chemical composition of fa can be found out either by X-ray fluorescence or by chemical titration method. Chemical composition of different plants of India is,

Table 2.1 Chemical composition of Indian fly ashes

Constituents	**Mohapatra et. al.**		**Valentim et. al.**			**Ogundiran et. al.**
SiO_2	57.00	56.00	61.54	57.86	62.21	62.1
Al_2O_3	29.28	33.07	26.94	24.57	2.56	27.4
Fe_2O_3	6.20	4.84	3.21	7.44	9.48	-
CaO	1.00	0.27	1.64	1.91	0.48	1.07
MgO	0.35	2.10	0.56	1.30	0.84	0.44
TiO_2	1.90	1.10	2.03	1.64	1.55	2.64
Na_2O	0.15	0.08	0.12	0.15	0.11	-
K_2O	0.41	0.12	1.78	1.99	1.28	0.83
P_2O_5	0.30	0.09	0.44	0.77	0.29	0.27
SO_4^{2-}	0.064	0.065	0	0.48	0.03	-
LOI	3.00	1.60	1.45	1.51	0.07	1.49

2.2 Phase Determination

X-ray Diffraction analysis use to determine the different type of phases present in fly ash. What type of phases present in fly ash and their determination is important to get idea about the combinations of the constituents of fly ash.

Ma et al. (2012) analysed the effect of alkali activation on fa using microstructure, mineralogy and mechanical strength. Geopolymerization of fa is done using NaOH and sodium silicate solution. Different amount of alkali solution was added and effect of these solutions is studied using X-ray diffraction and scanning electron spectrometry. It is stated that as the increase in solution, silica content decrease due to formation of zeolite crystalline.

Chand et. al. (2016) investigated properties of LD slag samples from 3 steel plants- Bokaro SP, Rourkela SP and Tata SP. Due to overlapping of peaks of different oxides present in the slag made difficult to understand phases present in it. Hazardous constituents were also determined using three different method and compared all slag samples on toxic limits.

2.3 Scanning electron microscopy and energy dispersive spectrometry

Kutchko et al. (2006) did detailed analysis on 12 clsss F fa which are taken from 9 different plants. Mineral characterisation was done using well known scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). Surface texture of samples and internal structures of particles were examined. Fly ashes have mainly two type of spherical surface. One is amorphous alumina-silicate spheres and other one is iron rich spheres. Iron rich spheres were consisting of oxide of iron and amorphous alumino-silicate. According to Kutchko, particle surface made from combination of of both materials and from the cross-section view, it is cleared that the FeO_2 and amorphous alumina-silicate were mixed all over the fa particles. Calcium is favourable to react with oxygen, phosphorus or sulphur and less reactive with silicon or aluminium. The calcium-rich material was distinct in both elemental composition and texture from the amorphous alumino-silicate spheres. In this analysis, primary mineral structure of the particle of fly ash are fairly common. Quartz present in crystalline form and alumina-silicate in amorphous form.

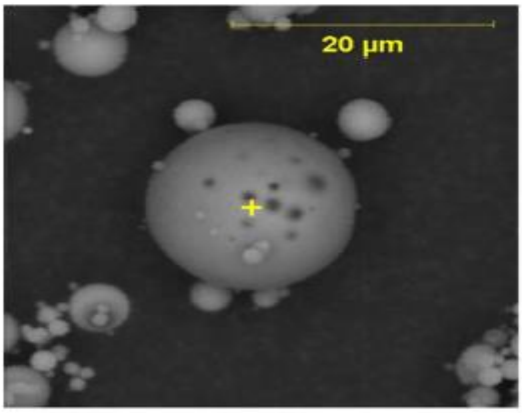

Figure 2.1 SEM image of class F fly ash (Kutchko et al.)

2.4 Loss on ignition

ASTM D 7348-08 is the method which is used to determine the loss on ignition of fa sample. In his method, fa heated to high temperature to find losses. These losses may be due to loss of carbon , sulphur, moisture or may be from decomposition of the residue. We can do test at to steps or in on steps and at two different temperature.

2.5 Free lime content

Free lime is important aspect to determine when fa is using with cement. It affect the properties of concrete and may cause the extension of the concrete which affect the total life of the structure.

Nawaz et al. (2015) performed tests on fa to determine the effect of free lime in cement concrete. Experimental results showed that higher free lime means less initial setting time, high compressive strength, and higher expansion of sample. Tests were done on fa and replacing cement with different amount of fa and adding variable amount of lime in concrete. It is concluded that free lime upto 4.23 % will not affect properties when fly ash replacement was 40% and it is 10% when fly ash replaced cement was 20%. These parameters were required to satisfy durability requirements.

2.6 Toxic Characteristics leaching procedure

Fa content little amount of trace elements which are very harmful for the environmental purpose. It may mix with ground water and affect the all living things. In Indian coal, amount of trace elements are very less. In fa, main toxic elements are As, Cr, Cu, Ni, Pb, Zn, Cd, Hg, Bi etc.

Mester et al. (1999) performed two different methods of acid digestion to find out the trace materials. Two acid digestion methods are microwave decomposition and wet acid digestion. In microwave digestion, chemical can be used in different combination. It is concluded that Nitric acid is strong enough to dissolve metals present in the fa but it is unable to solubilise silica particles which have maximum percentage in the fa. For this, hydrofluoric acid should be use. HF can dissolve silica particles easily. After doing acid digestion, toxic elements were find out using atomic absorption spectrometry. After studied different combination, acid digestion using HNO_3+HF+$HClO_4$ gave most efficient results.

Helal Uddin et al. (2016) also used different methods of acid digestion to find out toxic characteristic of the materials. This can be done on both organic and inorganic materials. Nitric acid is often use as a oxidising reagent. It may be used as individual or with other solvent. Different combination of solvent HCL, HNO_3 and $HClO_4$ had taken to acidify samples. Form the experimental result it can be concluded that combination of HNO_3-HCL in ratio of 1:3 was most efficient.

2.7 pH value

pH of the fly ash depends upon the type of coal it is extracted. Fly ash extracted after burning of subbituminous coal mostly gives class C fly ash. Fly ash with high amount of calcium oxide shows high alkalinity.

Cho et al (2005) studied about the removal characteristics of heavy metals from aqueous solution using fa. Class C fa is strongly alkali material with pH value around 10 to 13. Removal of heavy material like Pb, Cd, Zn depends upon the pH value of fa solution.

Roy et al. (2011) studied pH of the fa varying from strongly acidic fa to alkaline fa. It is concluded that acidic fa is buffered by dissolution of calcium and magnesium oxides and their pH increased with time up to 7-8. Alkaline fa buffered by mixing of carbon dioxide and pH reduced with time.

2.8 Alkali Activation of fly ash

Jaarsveld et al. (1999) studied about fa geopolymer and what is the effect of sodium and potassium present in the fa, on the setting time of geopolymer. To determine the rheological properties such as leaching of toxic minerals, strength of fa geopolymer, specific surface area and crystal structure of samples, different tests were done. It is concluded that activator used for geopolymerization affect each stage of formation of geopolymer. It affect from dissolution process of ions to crystal formation. Geopolymer immobilised the toxic elements after confined them in between tetrahedral structure.

Puertas et al. (2000) studied about activation of fa and slag pastes with sodium hydroxide. He took fa and slag in varying ratios (100/0, 70/ 30, 50/ 50, 30/ 70, and 0/ 100) and used NaOH of 2 and 10 M concentration. He cured the sample at 25°C and 65°C. from the study, it is concluded that as the compressive strength increased with slag percentage increment and amount of NaOH required for geopolymerization also affect the strength. Some of the fa particles dissolve in activator solution some will remain unreacted. The main compound form after reaction is a hydrated calcium silicate, like CSH gel. It present in its tetrahedral form and these tetrahedral formed polymer. Maximum strength came At higher concentration, i.e. 10M. Curing at elevated temperature of samples also affect the early strength gain. Strength gained by fa samples at 65 ^{o}C is much more that strength at room temperature.

Qureshi et al.(2014) studied alkali activated slag samples at different curing temperature to find physical properties such as strength, permeability and water absorption. The alkali-activation was done using with two type of solution in different combination. These solutions are KOH and sodium silicate. Curing had done on room temperature i.e. 28 ^{o}C, 40 ^{o}C, 50 ^{o}C and 60 ^{o}C. Combined alkali activators had taken which was 6.41%, 10.41% and 12.41% of the mass of blast furnace slag. The oven curing showed higher results compare to other activated slag samples. Similarly porosity and water absorption was also coming on sample which is oven cured.

Chapter 3

Methodology and material

3.1 Introduction

The materials used for construction of pavement should have some minimum criteria like it should have sufficient strength to withstand under traffic load. If it is required to utilise by-products in pavement construction, then it is required to know the properties of that material prior to the construction so that, we can predict the behaviour of by-products. Physico-chemical characterisation of fa was done according to codes and certain literatures.

3.2 Characterisation of fly ash

3.2.1 Titration method

Titration is used to determine different types of compound present in the fa sample. This method is easy to perform but we can only determine what type of oxides present in the sample FA like oxide of calcium, magnesium, silicon, iron and aluminium.

Figure 3.1 Titration of fly ash to find chemical composition

3.2.2 X-ray diffraction

The X-ray diffraction method is important to determine the phase composition of fa. Before doing the experiment, fa should pass from 75 microm sieve and it should be kept in over for 24 hr to dry. XRD analysis was performed on Rigaku, Ultima IV diffractometer using Cu Kα (λ=0.15405 nm) radiation. The diffraction data was taken in the scanning range (2θ range) of 0° to 100°, taking a scan speed of 20° per minute and a step size of 0.05. For analyse XRD data given by the machine, ‘X’pert high score software is required.

Figure 3.2 Rigaku Ultima IV diffractometer

3.2.3 Scanning electron microscopy (SEM) and electron dispersive spectroscopy (EDS)

The electron dispersive X-Ray spectroscopy is also a non-destructive, analytical technique used to determine the elemental composition of the materials under study. The elemental composition of a sample is determined using characteristic X-ray spectrum of the specimen being examined. The fa samples were analysed by means of scanning electron microscopy using a JEOL-JSM-6480 LV SEM. Platinum coating was done before performing the SEM test to protect it to get charged. The samples were put in the holder, and EDX spectrum of different points was observed. The element which have high atomic number reflect more brighter than the lower atomic number.

Figure 3.3 JEOL-JSM-6480 LV SEM

Figure 3.4 Platinum coating machine

3.2.4 Loss on ignition

ASTM D7348 procedure was used to run the LOI test. The method was performed with approximately 1 g of fa measured on analytical balance. In first step, sample was placed in cleaned crucible and heated at 110^{o} C in an oven for 1 hour. Samples took out from the oven after 1 hr, and put in a desiccator for 60 min before being reweighted. Sample loss in this step is recorded as moisture content in fly ash. In the second step, fly ash sample was placed in a furnace an air atmosphere and heated in a stepped schedule for 2 hour to reach to 950^{o} C. the fired sample was cool down at room temperature in a fully packed desiccator. After cooling down of samples again took weighted. The total weight loss after complete process gave the value of loss on ignition.

Figure 3.5 Fly ash samples after heated at 950oC in furnace

3.2.5 pH value determination

pH of the fly ash is determined as per IS:2720(xxvi)- 1987 ‘determination of pH'. According to this method, 30g of fly ash is had taken in 100 ml beaker and add 75 ml of deionised water. Stirrer the sample for few sec and then kept it for 1 hr with occasional stirring. After 1 hr, samples were tested using pH meter. Before using the pH meter, it should be calibrated using standard buffer solutions. The electrode was washed with distilled water and dried with ordinary filter paper and then it is immersed in fly ash suspension. Atleast two reading was taken and before taking reading, sample was properly stirred.

Figure 3.6 pH meter

3.2.6 Free lime content

Free lime content affect the properties of the concrete such as water requirement, setting time, compressive strength. Free lime content is determined using the method given in the literature. 1 gm of fly ash had taken and dissolve it into 50 cm^3 of ethylene glycol at the temperature of 60-70 ^{o}C for half hour period. Dissolution had done using magnetic stirrer. Than result solution was filtered from glass fibre filter paper.

3.2.7 Toxic characteristic leaching procedure (tclp)

Fly ash is a by-product of coal. It contents primary material as well as some trace materials. These trace materials are toxic which can affect the environment. Analysis of fly ash is done using an Atomic Absorption Spectrometer AAS. Toxic and hazardous materials determined by AAS compared with standard limiting data given by EPA. Some of the major toxic elements present in fly ash As, Cd, Cu, Fe, Pb, Zn, Ni and Hg. But before use the material in AAS, acid digestion is required. Acid digestion of the fly ash was done in a closed vessel device using temperature control microwave heating according to procedure provided in MILESTONE application book.

3.3 Physical properties of fly ash

3.3.1 Particle size analysis

Particle size Analysis had done according to IS 2720: Part 4: 1985 Methods of Test for Soils by using the hydrometer. The graphs are used to show the Particles Size Distribution. The results of this test are not presented here for publication point of view in future.

3.3.2 Specific gravity

Specific gravity of the fly ash was determined according to IS 2720 : Part 3 : Sec 1 : 1980 Methods of test for soils by using Le Chateliar flask with kerosene as a solvent. The values of specific gravity of fly ash are not presented here for publication point of view in future.

3.3.3 Optimum moisture content – maximum dry density relationship

The heavy compaction had done to determine compaction characteristics according to IS 2720 : Part 8 : 1983 Methods of Test for Soils. Moisture content was determined by using Sodium hydroxide solution of different percentages. Oven dry Fly ash was used. OMC and MDD is required to relate the moisture requirement and dry density.

3.3.4 Unconfined compressive strength

Compressive strength was determined according to IS 2720 : Part 10 : 1991 Methods of test for soils. Mould of size 38 mm diameter and 76 mm height was used to determine the strength of alkali activated fly ash at optimum moisture content and maximum dry density. Samples were cured at room temperature for 0 day, 7 days and 28 days. 0 day sample was tested within 2 hours after preparing the specimen.

Figure 3.7 proving ring for measuring load

3.3.5 California bearing ratio

CBR is a penetration test to evaluate the stability. CBR value of alkali activated fly ash was determined according to IS 2720: Part 16 : 1987 Methods of Test for Soil. Samples were prepared at optimum moisture content and maximum dry density. Sodium hydroxide solution was prepared before 24 hours. Samples were kept for 4 days under soaking condition. Before testing, specimen was allowed to drain downward for 15 minutes to remove free water collected in the mould.

Figure 3.8 CBR test for fly ash

3.4 Materials used

3.4.1 Fly ash

Class F type fly ash is used in this project work. This type of fly ash has low quantity of calcium oxide (CaO), so it does not have its own binding property. Fly ash is collected from 7 different sources-

3.4.2 Slag

Slag contained mainly lime, silica and alumina. It had taken from different sources-

3.4.3 Sodium hydroxide

Sodium hydroxide is used for alkali activation of fly ash. EMPLURA sodium hydroxide pellets with 97% pure NaOH was used as a alkali activator. Solution of NaOH of different percentage was kept for 24 hours before preparing the specimen.

3.4.4 Reagents

All the reagents use in different experiment on fly ashes are of AR grade.

Chapter 4

Results and discussion

4.1 Characterisation of fly ash

4.1.1 Chemical composition

Titration method is used to determine the percentage of oxides of silica, alumina, calcium, magnesium and ferrous present in fly ash.

The results of this test are not presented here for publication point of view in future.

4.1.2 X-ray diffraction

XRD was done to determine the phase composition of different type of fly ash. Data was analyse using the Philips X'pert high score software. The results of this test are not presented here for publication point of view in future.

4.1.3 Scanning electron microscopy

SEM had done to determine the particle morphology and surface structure of fly ash samples. Image of sample is taken from different position. The results of this test are not presented here for publication point of view in future.

4.1.4 Energy dispersive spectroscopy

EDS is used to determine particular atomic percentage. The results of this test are not presented here for publication point of view in future.

4.1.5 Loss on ignition

LOI test is basically used to determine the unburned carbon content in the fly ash. Water content and rheological property of fly ah concrete is affected by the amount of organic carbon present in it. The results of this test are not presented here for publication point of view in future.

4.1.6 Free lime content

Free lime content was determined of all samples. Free lime effect the behaviour of fa when we use in concrete construction. The results of this test are not presented here for publication point of view in future.

4.1.7 pH value

pH of different fa samples are determined as per IS 2720 part 26- 1987. The results of this test are not presented here for publication point of view in future.

4.2 Physical properties of fly ash geopolymer

4.2.1 Heavy compaction test

Optimum moisture content and maximum dry density of fa at different percentage of sodium hydroxide was determined. The results of this test are not presented here for publication point of view in future.

4.2.2 Unconfined compressive strength

Strength of fa was determined at different percentage of sodium hydroxide. The results of this test are not presented here for publication point of view in future.

4.2.3 California bearing ratio test

Specimen of alkali activated fa was prepared using maximum dry density and optimum moisture content. Specimen was kept under soaking condition for 4 days. Following values was obtained after testing the alkali activated fa. The results of this test are not presented here for publication point of view in future.

Chapter 5

Summery and future scope of the work

5.1 Summery

This study mainly comprised two sections. First section is focussing on the characterisation of fa and slag to know their chemical composition, shape, surface texture, unburnt carbon content, free lime content, toxic elements and pH value. In the second one, stabilisation of the fly ashes was done using alkali activator. Sodium hydroxide is used as alkali activator which is showing good results with fly ashes. This showed another way to use by-products in beneficial manner.

5.1.1 Characterisation of fly ash and slag

The results are not discussed here for publication point of view in future.

5.1.2 Stabilisation of fa using sodium hydroxide

The results are not discussed here for publication point of view in future.

Chapter 6

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